A Study of Ash Formation During the Combustion of Pulverized Coal

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INTRODUCTION

In order to predict the potential problems caused by ash in a pulverized coal in comparison to a coal-water fuel fired systems, it is necessary to understand the mechanism by which an ash particle acquires its particular size and compositional character. The particle size distribution (PSD) of an ash and its composition are determined by the following: the mineral matter composition and size distribution in the coal, the coal composition and particle size, the morphology of the char produced upon devolatilization of the coal particle, the local atmosphere surrounding the mineral particles, and the phase transformations of the mineral matter during combustion. When one or more of the above characteristics or conditions varies, the resulting ash size and composition may also change. In the process of preparing and burning a coal-water fuel, several or all of the above characteristics or conditions are different from those of the pulverized coal. It is the objective of this research to understand the effect that the form in which a coal is fired, pulverized versus coal-water fuel, has on the mechanisms responsible for the size and composition of the char and ash produced during combustion. Two coals, the Elk Creek biruminous and Beulah lignite, are being studied. Each coal is being fired in a pulverized form and as a coal-water fuel.

The following discussion concentrates on the preliminary results of the combustion tests on the Elk Creek and Beulah pulverized coals. The results presented show that the two pulverized coals produce distinctly different char-ash morphologies and sizes. The discussion provides comparisons between the coal PSD, the PSD of the mineral matter in the coal, and the PSDs of the char-ash samples collected at various locations down the combustor representing various degrees of burnout. The conclusions are based primarily on size data and observations of the morphologies of the various char-ash samples.

COMBUSTION SYSTEM AND ANALYTICAL TECHNIQUES

The combustion experiments were conducted in the down-fired combustor shown in Figure 1. The main radiant section of the combustor is modular and consists of four 0.46 m (18 inch) tall and 0.41 m (16 inch) diameter refractory sections. A divergent refractory cone, commonly termed a quart, is positioned on top of the circular refractory sections. The quart is 0.83 m (32.5 inches) high. The burner is located on top of the quart as shown in the Figure 1. The divergent cone top has a half-angle of approximately ten degrees and was used to minimize recirculation and swirt in the combustor. Swirt was not introduced in order to minimize ash deposition on the combustor walls and to ensure an even distribution of ash flow for sampling. Below the four circular refractory sections is a constrictor segment and a flue gas exit section. The flue gas passes through the convective section and enters a spray chamber to decrease the gas temperature prior to exiting the system via the induced draft fan and the stack. The overall length of the combustor is 3.05 m (10 feet). For more detail see Hurley (1990).

A series of 7.62 cm (3 inch) sampling ports extend the length of the combustor. Sample ports are numbered 1 through 10 starting at the top of the combustor. During this study, particulate and gas sampling were conducted in Ports 1, 2, and 10. Wall temperatures were monitored using thermocouples at eight locations along the length of the combustor. The temperature profile was considered stable when the temperature in the top four ports changed at a rate of less than 0.5°C/min. The combustor was preheated using natural gas at a firing rate of 0.26 GJ/h. The preheat period typically lasted approximately three hours after which the natural gas was shut off and the pulverized coal introduced. The fuels were fed at a rate of 0.32 GJ/h corresponding to a volumetric heat release rate of 1.14 GJ/m³-h. The Beulah lignite and Elk Creek bituminous pulverized coals were fed using an open helix dry feeder at a rate of 19.1 kg/h and 10.0 kg/h, respectively. The coal was entrained from the dry feeder and transported to the burner by the primary air which accounted for 18% of the total air required for combustion. The fuels were fired at 20% excess air. Total particle residence times within the radiant section of the combustor for the Beulah and Elk Creek coals were 2.09 and 2.13 seconds, respectively. Particle residence times for the samples collected at Ports 1, 2, and 10 are listed in Table 1. Overall carbon conversion levels of 99.4% and 99.9% were obtained at Port 10 for the Elk Creek coal and the Beulah pulverized coal, respectively.

During combustion, particulates were sampled isokinetically at a constant volumetric rate using a water-cooled sampling probe. A three-stage Anderson multicyclone and filter assembly was used to

classify the particulates during sampling. The cyclone aerodynamic diameter 50% cutpoints were 15 μm (cyclone 1), 2.7 μm (cyclone 2), and 0.46 μm (cyclone 3). The final filter was a polypropylene fiber filter which collected particles greater than 0.3 μm in diameter. Sampling isokinetically and at a constant volumetric rate ensured collecting representative samples of different sized char and ash particles by the probe and that the 50% cut sizes of the cyclones were consistent at each port. Gas sampling was conducted using a second water-cooled probe. The gas sample was withdrawn at each port and passed through a refrigeration unit to remove moisture from the gas prior to entering O_2 , CO, CO_2 , SO_2 , and NO_χ analyzers.

The ultimate analysis of each coal was determined using a Leco Corporation CHN-600 and a Leco Corporation SC-132 Sulfur Determinator. Sulfur forms of the coals were determined using ASTM D 2492. Calorific values of the coals were determined using a Parr Adiabatic Calorimeter. Proximate analysis of the coals was determined using a Leco Corporation MAC-400 proximate analyzer. Proximate analysis, ultimate analysis, and calorific value for both coals are presented in Table 2. The proximate analysis of the cyclone samples was determined thermogravimetrically with a Perkin Elmer 10 Series Thermal Analysis System. The thermogravimetric analyzer (TGA) was used for the proximate analysis of the cyclone samples because the TGA requires only 10 milligrams of sample for each analysis as compared to 1 gram for the MAC-400. TGA data was also used to determine the percent burnout, or carbon conversion, of the samples collected in the cyclones. Fuel burnout was calculated using ash as a tracer. Bulk inorganic compositional analysis for each coal ASTM ash was determined by a Spectrometrics Spectrospan 3 direct plasma spectrometer (DCP) (Table 2). The inorganic elements analyzed included silicon, aluminum, calcium, sodium, potassium, iron, magnesium, manganese, titanium, phosphorous, barium, sulfur, and strontium. Elemental analyses are reported on an oxide basis.

Computer-controlled scanning electron microscopy (CCSEM) was used to identify and quantify the size and composition of the mineral matter in the raw coal. The system is operated in conjunction with energy dispersive X-ray analysis provided by a Tracor Northern Electron Probe Microanalyzer EDS system at the University of North Dakota Energy and Environmental Center. The compositional analysis of each particle was compared with known compositional ranges of different mineral species. The computer then categorized each particle as a specific mineral phase. Mineral size data are presented in graphical form in the results section. Actual mineralogical analysis for each coal is not presented at this time. For a more detailed description of the sample preparation and procedure refer to Hurley (1990) and DeHoff and Rhines (1968). Photomicrographs were taken with a ISI ABT SX-40A SEM to study the morphology associated with the various cyclone sized fractions sampled at the different ports.

Size analysis of the coals and ash samples was conducted with a Malvern 2600 Particle Size Analyzer. The Malvern Particle Size Analyzer is an optical, nonimaging technique used to make in situ particle size measurements (Meyer, 1986) Composite samples from each sample port (i.e. Ports 1, 2, and 10) were used for size analysis. The particle size data are presented as cumulative volume percent as a function of the log size in microns. The data are presented in this manner in order to compare with the Malvern results which are reported on a volume distribution basis for each size category.

RESULTS AND DISCUSSION Relation Between Coal, Mineral Matter, and Char-Ash Particle Size Distributions Elk Creek Coal

In the case of the Elk Creek coal the data suggest that the individual mineral particles in the original coal do not form individual ash particles but rather that several mineral particles form an individual ash particle either by coalescence or agglomeration. The PSDs of the Elk Creek coal, mineral matter, and charash samples are shown in Figure 2. The d_{50} of the charash samples collected from Ports 1, 2, and 10 are 28.1,21.1 and 8.6 μ m while d_{50} of the coal is 19.4 μ m. The increase in particle size at Port 1 is seen throughout the entire size range. A possible explanation for the increase in PSD is that the Elk Creek coal has a Free Swelling Index (FSI) of 6 and exhibits swelling during heating. Such coal will typically pass through a fluid or plastic phase at approximately 350 to 400°C (Scaroni et al., 1986). During the fluid phase, volatiles are evolved which result in swelling of the coal particle.

Examination of SEM micrographs indicates that samples collected at Port 1 are composed of spherical char particles which have gone through a melt phase and experienced swelling. Holes due to the release of volatiles are visible on the char surfaces. The extent of carbon conversion is approximately 59.8%. Char cenospheres are present and fragmented, suggesting rapid heating rates. According to Solomon and Hamblen (1985), char particles melt and gas is evolved into the internal micropores forming small gas pockets. With continued heating, the size of the gas pockets increases and in some cases they may coalesce. At this point, significant swelling is not usually observed. According to Solomon and Hamblen (1985), the particle changes which subsequently occur are determined by the heating rate. With

rapid heating ($10^5\,^{\circ}\text{C/s}$), the cell walls rupture, blowing multiple holes in the surface of the char. At an intermediate heating rate, some coalescence of the bubbles occurs before rupturing. At lower heating rates (i.e. $10^3\,^{\circ}\text{C/s}$), the gas pockets expand and coalesce, eventually forming large cenospheres. In the second cyclone, ruptured char cenospheres containing individual and coalesced ash spheres are present which suggests the particles experienced rapid heating rates. The heating rate of the d_{50} ($19.4\,\mu\text{m}$) Elk Creek coal particle in the combustor is estimated to be approximately $3.0\,\times\,10^5\,^{\circ}\text{C/s}$. Based on the particle size data and the char morphologies, it is reasonable to suggest that the coarser size distribution of the samples collected at Port 1 is due to the swollen char particles found in the first cyclone.

At Port 2, the char particles are 88.9% burned out. The char cenospheres are lacy in appearance

At Port 2, the char particles are 88.9% burned out. The char cenospheres are lacy in appearance and numerous ash spheres are present on the larger char surfaces. The ash spheres appear on the char surface as the surface recedes during combustion. As the char surface recedes, mineral matter particles in the char are exposed at the char surface and melt forming slag beads on the char surface. However, individual slag beads do not coalesce or spread upon the char surface due to their inability to wet a carbon surface. Eventually, as the char surface continues to recede, the ash particles will coalesce forming larger solid ash particles. Some char cenospheres are fragmented either due to impact or rupture during gas

evolution.

At Port 10, the overall carbon conversion level is 99.4%. The PSD distribution of the sample collected at Port 10 approaches the PSD of the original mineral matter (Figure 3). In fact, the curves meet at approximately 48 μ m, however, the mineral matter PSD consists of much finer particles than the final ash sample. The data suggest that the individual mineral particles in the original coal do not form individual ash particles but rather that several mineral particles form an individual ash particle either by coalescence or agglomeration. If each individual mineral particle were responsible for forming an individual ash particle, the PSD curves of the ash and mineral matter would be nearly identical in shape. Evidence for the coalescence of mineral matter is based on the morphology of the char and ash during combustion and the composition and size of the mineral matter in the raw coal. The individual ash particles are composed of small ash spheres which are coalesced and highly agglomerated. The char only occurs as highly fragmented lacy structures (Figure 4).

The SEM evidence supports the premise that individual mineral particles do not form individual ash particles but rather several mineral particles in the form of coalesced ash spheres comprise an individual ash particle. The result is that the ash PSD at Port 10 consists of larger particles than the mineral matter

PSD in the raw coal.

Beulah Lignite

The PSDs of the Beulah coal, mineral matter, and char-ash samples are shown in Figure 4. The interpretation of the PSD data is that the char and the larger mineral matter particles in the coal undergo shedding and fragmentation during combustion. The d_{50} of samples collected from Ports 1, 2, and 10 decreases from 67.0 to 46.8 to 8.2 μ m as carbon conversion increases. However, the sample collected at Port 1 has a larger volume mean diameter than the original coal mean volume diameter of 46.4 μ m. The shift to a coarser PSD can be attributed to burning of the smaller coal particles thereby removing then from the total PSD. Unlike the Elk Creek coal, the Beulah coal has a FSI of 0. Therefore, the possibility of swelling as the mechanism responsible for the larger particle sizes is not reasonable. The carbon conversion at Port 1 is approximately 20%.

The samples collected from Port 2 show more extensive recession of the char surface. The surface of the char is sharply convoluted with ash cenospheres located between the ridges. The ash spheres are approximately $5 \, \mu m$ in diameter and are fairly uniform in size. The ash spheres represent mineral matter that has melted on the char surface as it is exposed by the receding char surface. The Beulah lignite does not pass through a fluid phase during heat up as does the Elk Creek coal and this may explain the difference in the surface appearance of the Beulah char as compared with the smooth surface of the Elk Creek char. As the char continues to burn and the char surface recedes, the char surface becomes even more jagged. Some fractured lacy carbonaceous particles and ash spheres are present in the smaller size fraction of the second cyclone where burnout is approximately 95.4%.

As with the Elk Creek coal, the size distributions for the Beulah samples collected from Port 1 through Port 10 shift to a finer overall PSD as would be expected with increasing burnout. However, unlike the Elk Creek ash PSD at Port 10, the Beulah ash PSD at Port 10 is characterized as finer than the mineral matter PSD in the original coal. The greatest difference between the PSD of the original mineral matter and the sample collected at Port 10 occurs at the coarser end of the cumulative volume percent curve. The curves actually superimpose in the size range of 4.6 µm and smaller. The interpretation of the PSD data is that the char and the larger mineral matter particles in the coal undergo shedding and fragmentation during combustion which results in the finer PSD of the ash relative to the mineral matter.

Therefore, individual large mineral particles and coal particles are actually forming several ash particles. Evidence for this is based on the morphology of the char and ash and the size and composition of the mineral matter in the pulverized coal. The mineral matter which is finely dispersed throughout the coal particle would normally form small, molten spheres on the char surface and eventually coalesce forming larger ash spheres. However, if char fragmentation occurs, the small mineral particles are unable to coalesce. The molten ash particles are released on smaller fragments of char and may coalesce but the overall increase in ash sphere size is minimized. The ash spheres may also be released into the gas stream as individual particles and retain their original size contributing to the finer size fraction of the ash. Fragmentation of ash agglomerates and cenospheres can also occur contributing to an increase in the smaller size fractions of ash. Fragmentation by either the ash particles separating from the char prior to char burnout or the fragmentation of ash cenospheres during and after char burnout are probably both occurring in the Beulah coal char and ash.

At Port 10, the overall carbon conversion is 99.88%. Micrographs of the composite sample show the ash to be composed of mostly separate individual angular ash and char particles, fragmented cenospheres, solid ash cenospheres (1.5 to 6.0 µm in diameter), and sintered spheres joined by neck structures (Figure 5). In general, there is no agglomeration of the larger ash particles suggesting that, as ash spheres are formed on the char surface they do not coalesce to form larger ash spheres or perhaps the

char fragments prior to the ash spheres coalescing.

The mineral matter composition and its PSD in the Beulah coal also provide an additional explanation for the finer PSD of the ash as compared to the coal mineral matter. Thirty-four percent of the mineral matter by weight is pyrite. Ninety-seven percent of the pyrite by weight is greater than 4.6 µm in diameter and 60% is greater than 48 µm in diameter. In addition, of the 32% of the total mineral matter in the Beulah coal that is greater than 48 µm, 14.2% is pyrite. The pyrite can be considered to be mostly extraneous based on its large size when compared to the pulverized coal PSD. Large particles of pyrite are known to fracture and disintegrate when rapidly heated (Helble et al., 1989a, 1989b; Baxter and Mitchell, 1989). Some of the pyrite fragments will agglomerate upon melting; however, the resulting particle sizes of the iron-containing species are generally still smaller than the original pyrite particles. Helble et al. (1989a, 1989b) investigated the effects of oxygen levels on pyrite oxidation and subsequent fragmentation during combustion of the Beulah lignite in a drop-tube furnace. Their results showed that the ash PSD was less than the mineral matter PSD in the presence of 21% oxygen. The results from this study are in direct agreement with Helble's results in that the Port 10 sample has a finer PSD than the coal mineral matter PSD. The fragmentation of larger sized mineral particles has also been noted by Srinivasachar et al. (1989) and Bryers (1985). The fragmentation occurs primarily in pyrite particles greater than $4.6\,\mu m$ based on the experimental PSD of the ash and mineral matter. Pyrite rapidly decomposes to pyrrhotite (Fe_{1-x}S) at 1027°C. In the presence of oxygen, pyrrhotite is converted to magnetite (Fe₃O₄). The final step in the oxidation process is the oxidation of magnetite to hematite (Fe₂O₃) (Helble et al., 1989a). Based on the evidence available at this time, the fragmentation of pyrite appears to contribute to the finer PSD of the final ash sample collected at Port 10 relative to the mineral matter in the original pulverized coal. Therefore, one larger mineral particle is actually forming several ash particles.

It should also be noted that 30% of the mineral matter in the Beulah coal was classified as unknown by the CCSEM. This mineral matter tends to be less than 2 µm in size and is composed of iron sulfur compounds and aluminosilicates. These particles are most likely finely disseminated throughout the coal particle and are therefore referred to as inherent. These mineral particles are not included in the PSD of the mineral matter shown in Figure 7. It is possible that these mineral particles may coalesce during combustion of the coal and appear as larger, although still relatively small ash particles in the PSD of the Port 10 sample. The result is an increase in the small particle portion of the PSD curve for the samples collected at Port 10 compared to the original mineral matter PSD. The fragmentation of the char and the larger extraneous mineral particles, and possible coalescence of disseminated mineral matter are the

predominating mechanisms determining the PSD of the final Beulah ash sample.

CONCLUSIONS

Based on the evidence, the different mechanisms responsible for the char-ash PSDs for the Elk Creek and Beulah coals are agglomeration and fragmentation, respectively. The chars and ash particles produced during the combustion of the Beulah and Elk Creek pulverized coals exhibit very different morphologies and PSDs relative to the original coal and mineral matter PSDs. For instance, the initial Elk Creek coal char samples collected at Port I were coarser than the original coal PSD due to swelling of char particles. The explanation for the increase in char PSD is based on the increased internal pressure generated by volatiles released during devolatilization while the coal is fluid causing increased swelling and the formation of large cenospheres. The result is a coarser PSD of the char samples collected at Port 1 than the original coal PSD.

The final Elk Creek ash PSD (Port 10) was coarser than that of the original mineral matter. Evidence based on particle sizing and SEM photomicrographs suggests that the individual mineral particles in the original coal do not form individual ash particles, but rather several mineral particles form an individual ash particle. The Elk Creek char passes through a fluid phase forming numerous carbonaceous cenospheres. Individual mineral matter particles form individual molten ash spheres on the exterior surface of the char as the char surface recedes during burning. The ash spheres gradually coalesce as the char surface continues to recede forming larger ash spheres. The end result is a coarser PSD for the final ash than the original PSD of the mineral matter.

A final ash PSD finer than the original mineral matter in the coal was observed during the Beulah testing. The particle size data and the SEM photomicrographs suggest that the char and the larger mineral matter particles in the coal undergo shedding and fragmentation during combustion which results in the finer PSD of the ash relative to the mineral matter. Therefore, individual large mineral particles and coal particles are actually forming several ash particles. Char samples collected from Port 1 have a fragmented appearance and the surface of the char particles tends to be very jagged. As the char surface recedes, mineral matter becomes exposed on the surface forming small ash spheres. Unlike the Elk Creek chars, the Beulah char often fragments prior to coalescing of the ash spheres. This char fragmentation results in

smaller ash particle sizes.

A second source of smaller ash particles is the pyrite in the coal. The larger pyrite fragments are not present within the coal particle but rather occur as extraneous particles. These pyrite particles often fragment when heated rapidly and oxidized during combustion and form numerous smaller iron-rich particles in the final ash (Srinivasachar et al., 1989 and Bryers, 1989). In addition, finely disseminated iron-rich particles and aluminosilicate particles are present throughout the coal particle. It is possible that these mineral particles may coalesce during combustion of the coal prior to char fragmentation and appear as larger, although still relatively small, ash particles in the ash PSD. The end result of both early char fragmentation and pyrite fragmentation, as well as the agglomeration of extremely fine mineral particles, is a finer ash PSD than mineral matter PSD in the original coal.

Based on the evidence, the different mechanisms responsible for the char-ash PSDs for the Elk Creek and Beulah coals are agglomeration and fragmentation, respectively. The d50 values of the Beulah and Elk Creek coals are quite different being $46.4\,\mu m$ and $19.4\,\mu m$, respectively and the mineral matter in the Beulah coal is coarser than that in the Elk Creek coal. However, the d_{50} values of the samples collected at Port 10 during the Beulah and Elk Creek tests are very similiar being 8.2 µm and 8.6 µm, respectively. The process of fragmentation results in the finer PSD of the Beulah ash relative to the coal mineral matter PSD. The process of coalescence and agglomeration of the Elk Creek char and ash results in a coarser ash PSD relative to the Elk Creek mineral matter PSD. The result is that the final PSDs of the two samples have similiar d50 values.

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TABLE 1. Particle Residence Times

	Residence Time in Combustor (sec)				
Port	Elk Creek Particle ($d_{50} = 19.4 \mu m$)	Beulah Particle ($d_{50} = 46.4 \mu m$)			
1	0.160	0.157			
2	0.417	0.410			
10	1.820	1.790			

TABLE 2. Elk Creek and Beulah Pulverized Coal Analysis

Coal:	Elk Creek		Beulah	
	A.R.ª	Dry	A.R.	Dry
Proximate Analysis, %wt.				
Moisture	0.90		29.99	44.05
Volatile Matter	30.19	30.50	30.98	44.25
Fixed Carbon	62.85	63.40	33.02	47.10
Ash	6.06	6.12	6.01	8.57
Ultimate Analysis, %wt.				
Moisture	0.90		29.99	
Carbon	80.95	81.69	45.71	65.29
Hydrogen	4.89	4.94	2.98	4.26
Nitrogen	1.58	1.59	0.68	0.97
Sulfur	0.68	0.69	0.66	0.94
Oxygen	5.84	5.89	13.97	19.96
Ash	6.06	6.12	6.01	8.57
Higher Heating Value, MJ/kg	31.0	33.6	16.5	25.2
(Btu/lb)	(13,350	(14,470)	(7,116)	(10,837)
Particle Size Distribution ^b , µm				
D (v, 0.9)		58.9		129.4
D (v, 0.5)		19.4		46.4
D (v, 0.1)		5.3		8.7
Ash Composition, %wt.				
SiO ₂		53.70		18.00
Al ₂ O ₃		30.00		9.96
TiÔ ₂		1.63		0.48
Fe ₂ O ₃		6.95		9.96
MgO		0.90		6.05
CaO ·		1.23		20.90
MnO		0.02		0.07
Na ₂ O		0.67		9.55
K ₂ O		1.93		0.43
SO ₃		0.90		20.60
P2O5		0.19		0.67
BaO		0.12		1.40
SrO		0.16		0.80
LOI		2.05		1.32
Total		100.55		99.19

^{*}As Received

^bThe D(v,0.9), D(v,0.5), and D(v,0.1) values are the particle sizes where, respectively, 90%, 50%, and 10% of the particles, by volume, are less than the indicated particle size.

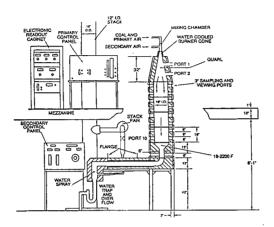


Figure 1. Down-fired Combustor

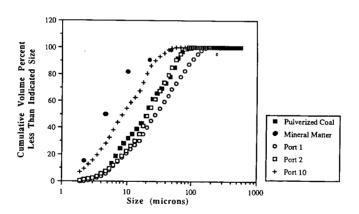


Figure 2. Comparison of PSDs of Elk Creek Coal, Mineral Matter, and Char-Ash Samples Collected from Ports 1, 2, and 10

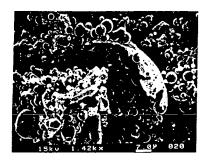


Figure 3. Elk Creek char-ash collected at Port 10.

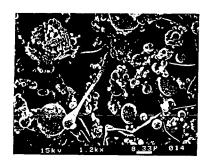


Figure 5. Beulah char-ash collected at Port 10.

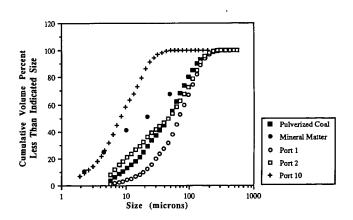


Figure 4. Comparison of PSDs of Beulah Coal, Mineral Matter, and Char-Ash Samples Collected from Ports 1, 2, and 10